

customer number 26645

PATENT

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

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IN THE APPLICATION OF

DOCKET No.: 3255

CRAIG D. TIPTON AND BILL A. WATERS

SERIAL No.: 10/752,894

EXAMINER: J. GOLOBOY

FILED: JANUARY 7, 2004

GROUP ART UNIT: 1714

TITLE: AUTOMATIC TRANSMISSION FLUIDS WITH PHTHALIC ACID CORROSION  
INHIBITION

Wickliffe, Ohio

Hon. Commissioner for Patents  
P. O. Box 1450  
Alexandria, VA 22313-1450

Sir:

**DECLARATION UNDER 37 C.F.R. §1.132**

I, Bill A. Waters, declare as follows:

I am a coinventor in the above-identified application and I am familiar with the references which were used in the rejection thereof.

I have been employed by The Lubrizol Corporation since 1968. I have been involved with chemical blending within the Research & Development Division for most of my career at Lubrizol. During the last 29 years, I have been the supervisor of the Blending Operations within R&D at Lubrizol.

Clarity of concentrates and blends is a key criteria for lubricant products. Lack of clarity or material "drop out" from a blend can suggest incompatibility or solubility issues. Through my experience in the blend lab I have gained expertise in the area of lubricant additives and their potential impact on blend clarity. I have been involved in various studies where blend order of components and blend conditions (temperature, mixing time

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on filed electronically on 8-27-07 By: Mary S. Didek

and agitation rate) were varied to investigate the impact of various variables on the solubilization of a blend.

In order to illustrate the improvement in performance of the compositions of the above invention, the following experiments were planned by Dr. Craig Tipton (another coinventor, since retired) and me and were performed by me:

Several lubricant formulations were prepared in mineral oil, containing various concentrations of terephthalic acid. The amounts of each component in the formulation are shown in the following Table. The formulations were prepared by one of two methods. The "Premixing" method involved mixing the terephthalic acid with an aliphatic phosphorus ester (dibutyl hydrogen phosphite) with heating to 130 °C, in the absence of other components or oil, and subsequently adding a succinimide dispersant. To the resulting mixture was added an inorganic phosphorus acid (phosphoric acid, 85%). The other listed ingredients were then added to complete the concentrate. The concentrate was then added at the appropriate treat rate to oil(s) to make the finished blend, as shown in the Table.

The other method, "No Premixing," involved adding the terephthalic acid to the mixture of the other components with no specific "premixing" of the terephthalic acid into any of the other components. This mixing was conducted at 80 °C, which falls within the typical temperature range used in blending concentrates (65-85C).

For the purpose of completeness, some additional formulations are reported in which the phosphoric acid was not present.

The resulting mixtures were evaluated for the solubility of the components in oil. Samples were stored for 1 week under the conditions indicated in the Table, below. The codes reported are for visual observation and characterization of the samples:

C = clear

VSLZ = very slight haze

SLZ = slight haze

Z = haze

T = trace sediment

L = light sediment

S = sediment

TABLE

| Formulation #                  | 110           | 115           | 111, 94        | 116, 132      | 112           | 117           |
|--------------------------------|---------------|---------------|----------------|---------------|---------------|---------------|
| Premixing:                     | —             | Yes           | —              | Yes           | —             | Yes           |
| Terephthalic acid              | <b>0.0012</b> | <b>0.0012</b> | <b>0.0025</b>  | <b>0.0025</b> | <b>0.0036</b> | <b>0.0036</b> |
| Di-butyl hydrogen phosphite    | 0.200         | 0.200         | 0.200          | 0.200         | 0.200         | 0.200         |
| Phosphoric acid (85%)          | 0.060         | 0.060         | 0.060          | 0.060         | 0.060         | 0.060         |
| Succinimide dispersant*        | 6.00          | 6.00          | 6.00           | 6.00          | 6.00          | 6.00          |
| Zinc dialkyl dithiophosphate*  | 5.34          | 5.34          | 5.34           | 5.34          | 5.34          | 5.34          |
| Ca sulf. detergent*            | 0.40          | 0.40          | 0.40           | 0.40          | 0.40          | 0.40          |
| <b>Solubility after 1 week</b> |               |               |                |               |               |               |
| RT with steel                  | C+T           | C             | C+T,<br>VSLZ+T | C, C          | C+T           | C             |
| 65 °C with steel               | C+T           | C             | C+T,<br>VSLZ+T | C, C          | C+T           | C             |
| 0°C, measured at 0°C           | C+T           | C             | C+T,<br>VSLZ   | C, C          | C+T           | C             |
| 0°C, measured at RT            | C+T           | C             | C+T,<br>VSLZ+T | C, C          | C+T           | C             |
| -18°C measured at -18°         | S             | S             | Z, Z           | S, S          | S             | S             |
| -18°C measured at RT           | C+T           | C             | C+T,<br>VSLZ+T | C, C          | C+T           | C             |

\*Amounts including conventional diluent oil

— No premixing

RT Room Temperature

TABLE, continued

| Formulation #                 | 113           | 118           | 114           | 119           | 95            | 133           |
|-------------------------------|---------------|---------------|---------------|---------------|---------------|---------------|
| Premixing:                    | —             | Yes           | —             | Yes           | —             | Yes           |
| Terephthalic acid             | <b>0.0048</b> | <b>0.0048</b> | <b>0.0064</b> | <b>0.0064</b> | <b>0.0125</b> | <b>0.0125</b> |
| Di-butyl hydrogen phosphite   | 0.200         | 0.200         | 0.200         | 0.200         | 0.200         | 0.200         |
| Phosphoric acid (85%)         | 0.060         | 0.060         | 0.060         | 0.060         | 0.060         | 0.060         |
| Succinimide dispersant*       | 6.00          | 6.00          | 6.00          | 6.00          | 6.00          | 6.00          |
| Zinc dialkyl dithiophosphate* | 5.34          | 5.34          | 5.34          | 5.34          | 5.34          | 5.34          |
| Ca sulf. detergent*           | 0.40          | 0.40          | 0.40          | 0.40          | 0.40          | 0.40          |
| <b>Solubility, 1 week</b>     |               |               |               |               |               |               |
| RT with steel                 | C+T           | C             | C+T           | C             | VSLZ+T        | C+T           |
| 65 °C with steel              | C+T           | C             | C+T           | C             | VSLZ+T        | C+T           |
| 0°C, measured at 0°C          | C+T           | C             | C+T           | C             | SLZ+T         | C+T           |
| 0°C, measured at RT           | C+T           | C             | C+T           | C             | SLZ+T         | C+T           |
| -18°C measured at -18°C       | S             | S             | S             | S             | S             | S             |
| -18°C measured at RT          | C+T           | C             | C+T           | C             | SLZ+T         | C+T           |

TABLE, continued

| Formulation #                 | 93     | 131   |  | 98     | 137    |  | 96     | 134    |
|-------------------------------|--------|-------|--|--------|--------|--|--------|--------|
| Premixing:                    | —      | Yes   |  | —      | Yes    |  | —      | Yes    |
| Terephthalic acid             | 0.050  | 0.050 |  | 0.0125 | 0.0125 |  | 0.0125 | 0.0125 |
| Di-butyl hydrogen phosphite   | 0.200  | 0.200 |  | 0.050  | 0.050  |  | 0.200  | 0.200  |
| Phosphoric acid (85%)         | 0.060  | 0.060 |  | 0.020  | 0.020  |  | 0      | 0      |
| Succinimide dispersant*       | 6.00   | 6.00  |  | 6.00   | 6.00   |  | 6.00   | 6.00   |
| Zinc dialkyl dithiophosphate* | 5.34   | 5.34  |  | 5.34   | 5.34   |  | 5.34   | 5.34   |
| Ca sulf. detergent*           | 0.40   | 0.40  |  | 0.40   | 0.40   |  | 0.40   | 0.40   |
| <b>Solubility, 1 week</b>     |        |       |  |        |        |  |        |        |
| RT with steel                 | VSLZ+T | C+T   |  | SLZ+T  | C+T    |  | C      | C+T    |
| 65 °C with steel              | C+T    | C+T   |  | VSLZ+T | C+T    |  | C      | C+T    |
| 0°C, measured at 0°C          | VSLZ+T | C+T   |  | Z      | C+T    |  | C      | C+T    |
| 0°C, measured at RT           | C+T    | C+T   |  | VSLZ+T | C+T    |  | C      | C+T    |
| -18°C measured at -18°C       | Z      | Z     |  | Z      | S      |  | Z      | S      |
| -18°C measured at RT          | VSLZ+T | C+T   |  | Z+L    | C+T    |  | C+T    | C+T    |

The results show that over a significant range of concentration of terephthalic acid, in the presence of phosphite ester and phosphoric acid, preparation of the lubricant composition by means of the pre-mixing procedure provides a product of improved clarity, that is, solubility of the terephthalic acid. This result is achieved at both relatively high and relatively low concentrations of the phosphite ester and the phosphoric acid. (The effect was not observed in the absence of the phosphoric acid: formulations 96 and 134. Additional formulations not reported in the Table, containing more than 0.05% terephthalic acid, did not exhibit good solubility by either route.)

I further declare that all statements herein made of my own knowledge are true and all statements herein made on information and belief are believed to be true. I understand that willful false statements and the like are punishable by fine or imprisonment or both (18 U.S.C. 1001) and may jeopardize the validity of the application or any patent issuing thereon.

Bill A. Waters  
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AUGUST 23, 2007  
date